PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2001-340718
(43)Date of publication of application : 11.12.2001

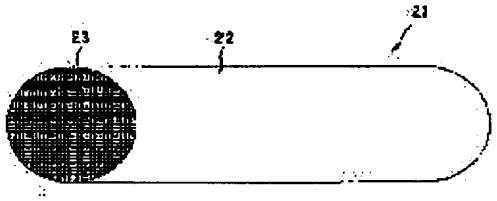
(51)Int.Cl. B01D 39/20
B01D 39/00
C04B 35/622
C04B 38/00

(21)Application number : 2000-165468
(22)Date of filing : 02.06.2000

(71)Applicant : NGK INSULATORS LTD
(72)Inventor : BABA TATSUO

(54) BASE MATERIAL FOR HONEYCOMB FILTER AND ITS MANUFACTURING METHOD

(57)Abstract:
PROBLEM TO BE SOLVED: To provide a base material for a honeycomb filter, which has a high mechanical strength and penetrate a large quantity of fluid.
SOLUTION: The base material 22 for the honeycomb filter is made of a porous body of honeycomb structure having a large number of cells 23. 50% particle diameter (D50) of aggregate particles constituting the base material 22 is made to be within a range of 40 to 100 μm. Further, >20 mass% and <80 mass% of the aggregate particles are constituted of globular particles of ≤1.1 aspect ratio and a residual part is constituted of nonglobular particles having aspect ratio of ≥ 1.2 times that of the globular particles.



CLAIMS

[Claim(s)]
[Claim 1]It is a substrate for honeycomb filters which consists of a porous body of honeycomb structure which has many cells, It is in within the limits whose 50% particle diameter (D_{50}) of an aggregate particle which constitutes the substrate concerned is 40-100 micrometers, And a substrate for honeycomb filters, wherein a 1.1 or less-aspect ratio spherical particle becomes [more than 20 mass% of aggregate particles concerned] in less than 80 mass % and, as for the remainder, an aspect ratio consists of a non-spherical particle of 1.2 times or more of said spherical particle.
[Claim 2]The substrate for honeycomb filters according to claim 1 with which particle size distribution of an aggregate particle fills relation between a following formula (1) and a following formula (2).
$0.7xD_{50}<=D_{20}$ -- (1)
$D_{80}<=1.3xD_{50}$ -- (2)
(However, D_{20}:20% particle diameter, D_{50}:50% particle diameter, D_{80}:80% particle diameter)
[Claim 3]A substrate for honeycomb filters with which pore volume distribution which is in within the limits whose 50% pole diameter (d_{50}) it is a substrate for honeycomb filters which consists of a porous body of honeycomb structure which has many cells, and is 5-25 micrometers, and was measured with a method of mercury penetration fills relation between a following formula (3) and a following formula (4).
$0.75xd_{50}<=d_{20}$ -- (3)
$d_{80}<=1.25xd_{50}$ -- (4)
(However, a d_{20}:20% pole diameter, a d_{50}:50% pole diameter, a d_{80}:80% pole diameter)
[Claim 4]It is a manufacturing method of a substrate for honeycomb filters including a process fabricated by extruding a plastic matter containing an aggregate particle from a cap for extrusion which has honeycomb structure and complementary shape, It is in within the limits whose 50% particle diameter (D_{50}) is 40-100 micrometers, And a manufacturing method of a substrate for honeycomb filters with which a ratio of a spherical particle whose aspect ratio is 1.1 or less is characterized by the remainder using a plastic matter which an aspect ratio prepared from an aggregate particle which consists of 1.2 or more times [of said spherical particle] the non-spherical particle in more than 20 mass % and less than 80 mass %.
[Claim 5]A manufacturing method of the substrate for honeycomb filters according to claim 4 which obtains a spherical particle by spray drying process.
[Claim 6]A manufacturing method of the substrate for honeycomb filters according to claim 4 or 5 with which particle size distribution of an aggregate particle which prepares a plastic matter fills relation between a following formula (1) and a following formula (2).
$0.7xD_{50}<=D_{20}$ -- (1)
$D_{80}<=1.3xD_{50}$ -- (2)
(However, D_{20}:20% particle diameter, D_{50}:50% particle diameter, D_{80}:80% particle diameter)
[Claim 7]A honeycomb filter which equipped cell inner skin of the substrate for honeycomb filters according to any one of claims 1 to 3 with at least one layer of filtration membranes with a small pole diameter 50% as compared with cell inner skin.

[Translation done.]

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]This invention relates to the substrate for honeycomb filters with a mechanical strength high in detail and large fluid transmission quantity about the substrate for honeycomb filters which consists of a porous body of the honeycomb structure which has many cells.
[0002]
[Description of the Prior Art]A honeycomb filter is a filter which uses as the substrate 22 the porous body of the honeycomb structure which has the cell 23 of a large number as shown, for example in drawing 1.
Since filtration is performed when processed fluids supplied to many cells 23, such as gas and a fluid, penetrate the fine pores of a porous body, it is used as a dust collecting filter with a large filtration area per unit volume, and a solid liquid separation filter.

[0003]For example, if the filter 21 is accommodated in housing and the peripheral face [of the substrate 22] and end face side is made into the structure isolated in airtight in O-ring etc., Only the filtration fluid which penetrated the inside of the fine pores of a substrate among the processed fluids supplied in the cell 23 can be made to be able to flow out of the peripheral face side, and the processed fluid which was not filtered can be used as a cross-flow filter collected from the end face side.
[0004]In a honeycomb filter, it is ideal to consider it as the structure which equips the inner skin of the cell 23 with at least one layer of filtration membranes (about 0.01-1.0 micrometer) whose pole diameter is still smaller as compared with the fine pores of a substrate, and to constitute the pole diameter inside a substrate greatly as much as possible. While securing a filtration efficiency by a filtration membrane with a small pole diameter in such a structure, it is because it is expectable that a pole diameter is large (1 - about 100 micrometers of numbers), and internal flow resistance makes fluid transmission quantity increase with a low substrate.
[0005]Generally, the substrate for honeycomb filters is manufactured by the method of fabricating by extruding the plastic matter containing an aggregate particle from the cap for extrusion which has honeycomb structure and complementary shape. As a method of obtaining a substrate with a large pole diameter, conventionally, ** By adding the method (henceforth "the 1st method") of enlarging the fine pores which are the interval parts between aggregate particles by enlarging the particle diameter of an aggregate particle, or the organic matters (a pitch, corks, etc.) which are burned down at the time of ** substrate calcination in a plastic matter, The method (henceforth "the 2nd method") of making a cavity part form and enlarging fine pores as compared with usual, etc. have been adopted.
[0006]
[Problem(s) to be Solved by the Invention]However, in the 1st method, there was a problem that the obtained substrate was not provided with the mechanical strength which can be equal to the use as a filter. In order to specifically damage by the handling in the process of producing a filtration membrane to the cell inner skin of a substrate, when a product yield fell or it was used as a filter, there was fault of damaging by the pressure of reverse washing.
[0007]Since an organic matter burned rapidly at the time of substrate calcination in addition to being hard to increase the amount of water penetration that a pole diameter cannot become large easily,

the 2nd method had the problem of producing a crack in a substrate by a thermal shock.
[0008]That is, in old, the substrate for honeycomb filters with a high mechanical strength and large fluid transmission quantity does not exist, but it is anxious for such a substrate. There is a place which this invention is made in view of the problem of such conventional technology, and is made into the purpose in providing the substrate for honeycomb filters with a high mechanical strength and large fluid transmission quantity.
[0009]
[Means for Solving the Problem]As a result of this invention persons' inquiring wholeheartedly, by controlling the 50% particle diameter of an aggregate particle and a mass ratio of a spherical particle which constitute a substrate within the limits of predetermined, it thought out for a problem of conventional technology to be solvable, and this invention was completed.
[0010]Namely, according to this invention, it is a substrate for honeycomb filters which consists of a porous body of honeycomb structure which has many cells, It is in within the limits whose 50% particle diameter (D_{50}) of an aggregate particle which constitutes the substrate concerned is 40-100 micrometers, And a substrate for honeycomb filters, wherein a 1.1 or less-aspect ratio spherical particle becomes [more than 20 mass% of aggregate particles concerned] in less than 80 mass % and, as for the remainder, an aspect ratio consists of a non-spherical particle of 1.2 times or more of said spherical particle is provided.
[0011]As for the above-mentioned substrate for honeycomb filters, it is preferred that particle size distribution of an aggregate particle fills relation between a following formula (1) and a following formula (2).
$0.7xD_{50}<=D_{20}$ — (1)
$D_{80}<=1.3xD_{50}$ — (2)
(However, D_{20}:20% particle diameter, D_{50}:50% particle diameter, D_{80}:80% particle diameter)
[0012]According to this invention, it is a substrate for honeycomb filters which consists of a porous body of honeycomb structure which has many cells, A substrate for honeycomb filters with which pore volume distribution which is in within the limits whose 50% pole diameter (d_{50}) is 5-25 micrometers, and was measured with a method of mercury penetration fills relation between a following formula (3) and a following formula (4) is provided.
$0.75xd_{50}<=d_{20}$ — (3)
$d_{80}<=1.25xd_{50}$ — (4)
(However, a d_{20}:20% pole diameter, a d_{50}:50% pole diameter, a d_{80}:80% pole diameter)
[0013]It is a manufacturing method of a substrate for honeycomb filters including a process fabricated by extruding a plastic matter containing an aggregate particle from a cap for extrusion which has honeycomb structure and complementary shape according to this invention, It is in within the limits whose 50% particle diameter (D_{50}) is 40-100 micrometers, And a manufacturing method of a substrate for honeycomb filters with which a ratio of a spherical particle whose aspect ratio is 1.1 or less is characterized by the remainder using a plastic matter which an aspect ratio prepared from an aggregate particle which consists of 1.2 or more times [of said spherical particle] the non-spherical particle in more than 20 mass % and less than 80 mass % is provided.
[0014]In a manufacturing method of this invention, it is preferred to obtain a spherical particle by spray drying process, and it is preferred that particle size distribution of an aggregate particle which prepares a plastic matter fills relation between a following formula (1) and a following formula (2).
$0.7xD_{50}<=D_{20}$ — (1)
$D_{80}<=1.3xD_{50}$ — (2)
(However, D_{20}:20% particle diameter, D_{50}:50% particle diameter, D_{80}:80% particle diameter)
[0015]According to this invention, a honeycomb filter which equipped cell inner skin of the above-mentioned substrate for honeycomb filters with at least one layer of filtration membranes with a small pole diameter 50% as compared with cell inner skin is provided again.
[0016]
[Embodiment of the Invention]The substrate for honeycomb filters of this invention controls the 50% particle diameter of an aggregate particle and the mass ratio of a spherical particle which constitute

a substrate within the limits of predetermined. The substrate for honeycomb filters of this invention has a high mechanical strength, and its fluid transmission quantity is large. Hereafter, this invention is explained in detail.

[0017](1) The substrate for honeycomb filters of substrate this invention (only henceforth a "substrate") makes at least one sort in an aggregate particle the spherical particle whose aspect ratio is 1.1 or less. By making such a spherical particle into an aggregate particle, after substrate calcination, an interval part (namely, fine pores) is certainly formed between aggregate particles, and transmission quantity of a fluid can be enlarged. By including said spherical particle in an aggregate particle as for more than 20 mass %, fluid transmission quantity is made to increase and, specifically, the effect which equalizes the microstructure inside a substrate can be acquired.

[0018]However, if all the aggregate particles that constitute a substrate are made into the above-mentioned spherical particle, combination between aggregate particles will become weak and the mechanical strength of a substrate will fall. Therefore, in addition to using more than 20 mass % of an aggregate particle, said spherical particle needs to make the maximum 80 mass %.

[0019]About the remainders other than a spherical particle, an aspect ratio constitutes among aggregate particles by 1.2 or more times [of the above-mentioned spherical particle] the non-spherical particle. That is, if it is a case where the thing of 1.2 or more and 1.1 is used when the particles of the aspect ratio 1.0 are used as a spherical particle, the particles which have 1.32 or more aspect ratios will turn into a non-spherical particle. Although a non-spherical particle is not preferred from a viewpoint of making fluid transmission quantity increase, it has an effect which strengthens combination between aggregate particles, and there is an operation which raises the mechanical strength of a substrate.

[0020]The average value of the aspect ratio (ratio of a long side to a shorter side) of 20 aggregate particles arbitrarily chosen from the photograph taken with the scanning electron microscope in powdered voice with the "aspect ratio" said to this invention, In a sintered compact, it is the average value of the aspect ratio similarly computed about the field which applied and made up for resin (for example, epoxy resin) to the cutting plane of the sintered compact, and also performed mirror finish.

[0021]It is required the range of predetermined in 50% particle diameter (D_{50}) and to specifically be controlled within the limits of 40-100 micrometers in addition to the spherical particle and the non-spherical particle filling the aspect ratio as stated above. In the point that fluid transmission quantity decreases when 50% particle diameter (D_{50}) is less than 40 micrometers, when set to more than 100 micrometers, it has set at the point that the mechanical strength of a substrate falls, and a gap is not preferred, either. The 50% pole diameter (d_{50}) of a substrate is also controlled within the limits of 5-25 micrometers (method of mercury penetration) by carrying out particle diameter (D_{50}) within the limits of 40-100 micrometers 50%.

[0022]The "x % particle diameter" told to this invention is particle diameter measured by the sifting-out method in powdered voice. That to which several screens from which the diameter of a nominal opening differs were specifically accumulated on multistage as the upper row so that the diameter of an opening might become large is prepared, After pouring into the screen of the highest rung the powder sample which is a measuring object of particle diameter and shaking for 15 minutes with a shaker, the particle size distribution curve was created from the relation between the powder mass in the plus sieve of each stage, and its diameter of an opening, and the particle diameter from which addition mass will be x % was specified as particle diameter x%.

[0023]As for a spherical particle and a non-spherical particle, it is preferred that particle size distribution is controlled by the predetermined range in addition to filling a spherical particle ratio as stated above and 50% particle diameter (D_{50}) and to specifically fill the relation between a following formula (1) and a following formula (2).

$0.7 \times D_{50} <= D_{20}$ — (1)

$D_{80} <= 1.3 \times D_{50}$ — (2)

(However, D_{20}:20% particle diameter, D_{50}:50% particle diameter, D_{80}:80% particle diameter)

[0024]It is because the particle size distribution of an aggregate particle becomes sharp [the substrate which fills the relation between the above-mentioned formula (1) and the above-mentioned formula (2) / pore volume distribution]. Specifically, the pore volume distribution measured with the

method of mercury penetration serves as a substrate controlled within limits which fill the relation between a following formula (3) and a following formula (4).

$0.75xd_{50}<=d_{20}$ -- (3)

$d_{80}<=1.25xd_{50}$ -- (4)

(However, a d_{20}:20% pole diameter, a d_{50}:50% pole diameter, a d_{80}:80% pole diameter)

[0025]On the other hand, when not filling the above-mentioned formula (1), it originates in a particle ingredient increasing and the cavity part between the aggregate particles of a substrate is blockaded, and there is a possibility that fluid transmission quantity may decrease. Since the slurry for film production enters into the cavity part concerned and blockades in a film production process although the cavity part between aggregate particles becomes large in not filling the above-mentioned formula (2), the fluid transmission quantity of a filter has too a possibility of falling.

[0026]The "x % pole diameter" said to this invention is a pole diameter measured with the method of mercury penetration which makes a following formula (5) a principle type. If mercury is pressed fit sequentially from fine pores with a large path, the accumulation capacity of mercury increases, if mercury is pressed fit specifically raising a pressure gradually to the dry substrate, and all the fine pores are eventually filled with mercury, accumulation capacity will reach the amount of **. In this invention, the pole diameter d computed from the pressure P at the time of accumulation capacity becoming x % was specified as the pole diameter x%.

d=-gammaxcos theta/P -- (5)

(However, d:pole diameter, gamma:surface tension, theta:angle of contact, P: Pressure)

[0027](2) The substrate for manufacturing method honeycomb filters is fabricated by extruding the plastic matter containing an aggregate particle from the cap for extrusion which has honeycomb structure and complementary shape, and is obtained by drying and calcinating the Plastic solid concerned. What is necessary is just to use an aspect ratio, 50% particle diameter, and the aggregate particle that controlled particle size distribution to previous statement within the limits, when preparing a plastic matter in order to manufacture the substrate of this invention.

[0028]Namely, it is in within the limits whose 50% particle diameter (D_{50}) is 40-100 micrometers, And as for the remainder, in more than 20 mass % and less than 80 mass %, the ratio of the spherical particle whose aspect ratio is 1.1 or less prepares a plastic matter from the aggregate particle which consists of a non-spherical particle whose aspect ratio is 1.2 or more times of said spherical particle. It is made for the particle size distribution of the aggregate particle concerned to fill the relation between a following formula (1) and a following formula (2) to make pore volume distribution of a substrate sharp.

$0.7xD_{50}<=D_{20}$ -- (1)

$D_{80}<=1.3xD_{50}$ -- (2)

(However, D_{20}:20% particle diameter, D_{50}:50% particle diameter, D_{80}:80% particle diameter)

[0029]What ground and classified remaining as it is or this, for example for the commercial ceramic raw material as the method of preparation is made into an aggregate particle, and the method of mixing suitably so that the conditions of previous statement of two or more sorts of aggregate particles may be fulfilled, etc. are mentioned.

[0030]In the manufacturing method of this invention, it is preferred to obtain a spherical particle by spray drying process. Although a spherical particle may use what was prepared by the method of grinding and mixing with a grinder or mixers (ball mill etc.), it is because a 1.1 or less-aspect ratio spherical particle is obtained comparatively easily according to the spray drying process which carries out the granulation and desiccation of the raw material made liquefied with a spray dryer, and calcinates it. Since it is smooth in the surface as compared with the particles obtained by grinding, the particles obtained by spray drying process do not hurt their cap for extrusion molding, but the life time is preferred also in the point which becomes long at about 10 times.

[0031]When the manufacturing method of this invention prepares a plastic matter, it can be conventionally manufactured by the publicly known manufacturing method and the same method except for using an aspect ratio, 50% particle diameter, and the aggregate particle that controlled particle size distribution by the request to previous statement within the limits.

[0032]A plastic matter adds an inorganic bonding material, a surface-active agent, a plasticizer, etc.

by carrier fluid, an organic binder, and necessity besides an aggregate particle, kneads, and is taken as a molding raw material.
[0033]As an aggregate particle, water etc. can be used as carrier fluid and alumina, mullite, SERUBEN, cordierite, silicon carbide, or these mixtures can be used for methyl cellulose etc. as an organic binder.
[0034]An inorganic bonding material is the add-in material for strengthening combination of an aggregate particle, and one sort or two sorts or more of mixtures of alumina with a particle diameter of less than 1 micrometer, silica, zirconia, a titania, glass frit, feldspar, and the cordierites can be used for it. Although an inorganic bonding material is a ceramic particle, it is not included by the aggregate particle said to this invention.
[0035]As for an inorganic bonding material, when mass of an aggregate particle is made into 100 mass %, it is [more than 15 mass %] preferred to add the quantity equivalent to below 35 mass %. While the intensity of a substrate falls that it is less than 15 mass %, it is because an inorganic bonding material stops at the gap of the aggregate particle of what is obtained, so sufficient intensity has a possibility of blockading the fine pores inside a substrate and reducing fluid transmission quantity when it becomes more than 35 mass %.
[0036]The substrate of honeycomb structure can be manufactured by carrying out extrusion molding of the plastic matter to desired shape, and drying and calcinating it. For example, a Plastic solid can be acquired by extruding the plastic matter supplied to conventionally publicly known extrusion machines, such as a screw extruder of a monopodium, biaxial, or a multiple spindle, and a plunger extruder, from the cap for extrusion which has the honeycomb structure of a substrate, and complementary shape.
[0037]It is possible to consider it as the end face form (circular, a square, a rectangle, a hexagon, etc.) of a substrate, an end face outer diameter (30-200mmphi when circular), the shape (circular, a quadrangle, a hexagon, etc.) of a cell, and the shape of a request of the inscribed hole diameter (usually 2-5 mmphi grade) of a cell, etc. with the shape of a cap. Although the size in particular of a substrate is not limited, that whose overall length of a longitudinal direction is about 150-2000 mm is used widely.
[0038](3) After making the slurry for film production containing an aggregate particle adhere to the cell inner skin of the filter above-mentioned substrate, a filtration membrane can be formed in it by the method of drying and calcinating the film production object concerned, and a honeycomb filter can be obtained to it.
[0039]Distribute an aggregate particle in carrier fluid, such as water, and if needed For example, an organic binder, It is considered as the slurry for film production by adding a pH adjuster, a surface-active agent, etc., Conventionally, it can form membranes and dry in cell inner skin using a publicly known method, for example, the dip producing-film method, the filtration producing-film method given in JP,63-66566,B which these people already indicated, etc., and a filter can be obtained by the method of calcinating the film production object concerned at an about 1300 ** elevated temperature further. The same thing as a substrate can be used about an aggregate particle, carrier fluid, and an organic binder. However, as for the 50% particle diameter of an aggregate particle, in order to make the pole diameter of a filtration membrane small, it is common to make it smaller than a substrate.
[0040]The slurry for film production may be made to contain an inorganic bonding material for the same purpose as a substrate. the case of a filtration membrane -- clay with a particle diameter of less than 1 micrometer, kaolin, and a titania -- sol and silica -- it is preferred that can use sol, glass frit, etc. and more than 5 mass % is contained by the ratio below 25 mass % in an aggregate particle and the total mass of an inorganic bonding material.
[0041]Although necessity has carried out 1 stratification at least, a filtration membrane is formed more than two-layer, and is good also as a double layer.
[0042]
[Example]Hereafter, this invention is not limited by the following example although an example explains the filter of this invention still in detail.
[0043]As a ceramic raw material used as the aggregate particle of a substrate, alumina (A1-A6), the mullite (M1-M3), and SERUBEN (S1) which have a presentation and aspect ratio of a statement were used for Table 1. These raw materials were mixed by the ratio of the statement to Tables 2-3, and the aggregate particle was prepared.

[0044]About A3 and M1, after making a raw material liquefied, a granulation and desiccation were carried out with the spray dryer, and the aspect ratio was made or less into 1.1 by calcinating.
[0045]
[Table 1]

記号	種類	粒度分布					アスペクト比	
		D50×0.7 μm	D20 μm	D50 μm	D80 μm	D50×1.3 μm	平均	1.1以下粒子 %
A1	アルミナ	60	62	85	106	111	1.8	0
A2		49	55	70	86	91	1.9	0
A3		53	65	75	85	98	1.1	100
A4		74	92	105	135	137	1.8	0
A5		18	18	26	33	34	1.8	0
A6		53	41	75	105	98	1.7	0
M1	ムライト	53	65	76	85	99	1	100
M2		46	45	65	87	85	1.5	0
M3		55	62	78	104	101	1.8	0
S1	セルベン	49	54	70	87	91	1.8	0

[0046](Substrate) Extrusion molding of the plastic matter which added and kneaded methyl cellulose to the above-mentioned aggregate particle as an organic binder besides inorganic bonding materials (feldspar, glass frit, etc.) and water was carried out, and the extrusion molding body of the honeycomb structure which has 37 cells (outer diameter phi30mm and diameter phi2.4mm) was obtained. The inorganic bonding material added the quantity which is equivalent to 25 mass % to this, when mass of an aggregate particle was made into 100 mass %. The substrate was obtained by calcinating the extrusion molding body concerned at 1500 ** with an electric furnace.
[0047]The above-mentioned substrate was evaluated about the 50% pole diameter of a substrate and pore volume distribution, and a mechanical strength.
[0048]About the 50% pole diameter and pore volume distribution of the substrate, it measured by the following methods in accordance with the method of mercury penetration. First, only a length of 25 mm cut down the substrate from the end face, and also it cut so that 4-5 cells might remain, it was considered as the sample for measurement, where the sample concerned is immersed into mercury, mercury was pressed fit, and a pole diameter, a 50% pole diameter, and an 80% pole diameter were computed 20% by measuring the accumulation capacity.
[0049]The mechanical strength of the substrate was evaluated by examining three point bending intensity by making into the distance between fulcrums of 80 mm the sintered compact which dries on the same conditions as a substrate, and calcinates the Plastic solid which extruded each plastic matter for shaping to cylindrical shape with a diameter phi20-mmx length of 100 mm.
[0050]The above-mentioned substrate formed the filtration membrane and evaluated it also about the performance as a filter. The filtration membrane was made into multiple layer structure, and provided the interlayer and the filter layer.
[0051]Film production of the interlayer and the filter layer was performed to JP,61-238315,A by the filtration forming-membranes method of the statement. As opposed to the device which specifically consists of the vacuum chamber 6 shown in drawing 2, the storage kettle 8, the pump 7, the flanges 2 and 3, and piping 10 grade, The cell inner skin [of the substrate 1 which replaced the inside of fine pores with fluids, such as water,], and substrate 1 peripheral-face side is fixed in the state where it isolated in airtight with the flanges 2 and 3 and the bolt 5, Subsequently, sending the slurry 9 in the storage kettle 8 continuously in the cell of the substrate 1 with the pump 7, and making the cell inner skin 12 contact, evacuation of the inside of the vacuum chamber 6 is carried out with the vacuum pump 13, and the substrate 1 peripheral-face side is made into a reduced pressure state. Since filtration differential pressure is given by such operation between a substrate 1 peripheral-face side and the cell inner skin 12 side, a slurry is produced by the cell inner skin 12 of the substrate 1, and the moisture in a slurry is discharged from the substrate 1 peripheral-face side as filtrate by it.
[0052](Interlayer) The aggregate particle, the inorganic bonding materials (feldspar, glass frit, etc.), and water whose particle diameter (D_{50}) is 3.2 micrometers were mixed with the substrate with the mass ratio of 27:3:70 50% with same material, and the slurry was prepared. After producing a slurry to

the inner skin of each cell, it dried, and by calcinating, it was made to adhere to a substrate and the interlayer was formed.

[0053](Filter layer) The aggregate particle, the inorganic bonding material (glass frit), and water whose particle diameter (D_{50}) is 0.4 micrometer were mixed with the substrate with the mass ratio of 9:1:90 50% with same material, and the slurry was prepared. After producing a slurry on the surface of the interlayer of each cell, it dried, and by calcinating, the interlayer was made to adhere and the filter layer was formed.

[0054]About the filter manufactured as mentioned above, the amount of water penetration estimated fluid transmission quantity, and the filtration efficiency was evaluated by the maximum pole diameter of the filter layer. After the amount of water penetration deaerates the air bubbles in a filter under underwater and decompression of 6.7 or less kPa for 2 hours, Pure water was injected into the cell of a filter on differential pressure 4.8 - 9.8kPa, and conditions with a temperature of 25 **, and it filtered by making it penetrate from the inside of a cell to the filter peripheral face side, and evaluated by measuring the amount of water penetration per time per filtration area.

[0055]About the maximum pole diameter of the filter layer, it measured by the following methods. The pressurized air was sent in from cell inner skin to the filter which carried out humidity to ASTM F316 with water with a water temperature of 20 ** based on the air flow method of a statement, raising a pressure gradually, and the pole diameter D computed from the pressure P by which air bubbles were checked by the beginning from the substrate peripheral face was made into the maximum pole diameter of a filter layer.

[0056](Example 1) The example which produced the substrate by having made into the aggregate particle the alumina particle which has various spherical particle ratios, 50% particle diameter, and particle size distribution as Example 1, and also formed the filtration membrane, and was used as the filter is shown.

[0057]

[Table 2]

	組成					骨材粒子								基材							濾過層		濾過層
	骨材1 種類		骨材2 種類			D50×0.7	粒度分布 D20	D50	D80	D50×1.3	アスペクト比1.1以下粒子原料中比率	d50×0.75	平均細孔径 d20	d50	d80	d50×1.25	曲げ強度	透水量	骨材粒子D50 中間層	濾過層	最大細孔径
		%		%		μm	μm	μm	μm	μm	%	μm	μm	μm	μm	μm	MPa	m3/hr*m2	μm	μm	μm
比較例1-1	A1	0	A3	100		53	65	75	85	96	100	12.2	13.9	16.3	18.4	20.4	16.5	72.7	-	-	-
比較例1-2	A1	20	A3	80		53	64	75	86	96	80	12.4	13.9	16.5	18.7	20.7	16.7	72.7	-	-	-
実施例1-1	A1	25	A3	75		53	63	76	88	96	75	12.5	13.9	16.7	19.6	20.8	33.1	73.6	3.6	0.7	<1.8
実施例1-2	A1	50	A3	50		54	62	77	82	100	50	12.7	13.7	17.0	20.2	21.2	32.5	75.0	3.6	0.7	<1.8
実施例1-3	A1	75	A3	25		55	62	78	87	101	25	13.0	13.0	17.3	21.2	21.7	31.9	76.2	3.6	0.7	<1.8
比較例1-3	A1	80	A3	20		55	61	78	101	101	20	13.2	13.2	17.6	22.9	22.0	31.9	76.2	3.6	0.7	4.2
比較例1-4	A2	20	A3	80		52	63	74	85	96	80	12.1	13.5	16.1	18.3	20.1	19.3	71.5	-	-	-
実施例1-4	A2	25	A3	75		52	62	74	86	96	75	12.0	13.3	16.0	18.9	20.0	34.3	71.5	3.6	0.7	<1.8
実施例1-5	A2	50	A3	50		51	60	73	86	95	50	11.7	12.7	15.7	18.4	19.6	34.8	70.3	3.6	0.7	<1.8
実施例1-6	A2	75	A3	25		50	57	71	87	92	25	11.2	11.7	15.0	18.0	18.7	36.0	68.0	3.6	0.7	<1.8
比較例1-5	A2	80	A3	20		49	56	70	87	91	20	11.0	11.4	14.7	18.4	18.4	36.6	66.8	3.6	0.7	2.8
比較例1-6	A4	20	A3	80		53	67	76	95	99	80	12.8	14.9	17.1	21.2	21.4	18.1	73.8	-	-	-
実施例1-7	A4	25	A3	75		55	70	78	99	101	75	13.0	15.5	17.3	20.8	21.7	31.9	76.2	3.6	0.7	<1.8
実施例1-8	A4	50	A3	50		62	72	88	113	114	50	15.5	16.7	20.7	24.8	25.8	26.1	87.8	3.6	0.7	<1.8
比較例1-7	A4	75	A3	25		71	77	102	125	133	25	18.0	18.8	25.3	30.6	31.7	17.9	104.2	-	-	-
比較例1-8	A4	80	A3	20		74	85	105	128	137	20	19.7	20.8	26.3	32.2	32.9	18.2	107.7	-	-	-
比較例1-9	A5	20	A3	80		51	30	73	83	95	80	11.9	9.4	15.9	18.7	19.9	19.6	70.3	-	-	-
比較例1-10	A5	25	A3	75		50	26	72	80	94	75	11.5	8.1	15.3	19.6	19.2	35.4	69.2	3.6	0.7	3.8
比較例1-11	A5	50	A3	50		29	20	41	88	53	50	6.2	5.8	8.3	9.1	10.4	53.5	33.0	-	-	-
比較例1-12	A5	75	A3	25		19	19	27	63	35	25	4.1	3.9	5.5	6.5	6.9	61.7	16.7	-	-	-
比較例1-13	A5	80	A3	20		18	18	25	35	33	20	4.0	3.6	5.3	6.7	6.6	62.8	14.3	-	-	-
比較例1-14	A6	20	A3	80		53	63	75	86	96	80	12.1	13.3	16.1	18.3	20.1	18.7	71.1	-	-	-
実施例1-9	A6	25	A3	75		53	61	75	86	96	75	12.3	13.2	16.4	19.5	20.5	33.4	70.2	3.6	0.7	<1.8
実施例1-10	A6	50	A3	50		53	55	75	92	96	50	12.2	12.3	16.2	19.8	20.3	33.1	69.8	3.6	0.7	<1.8
実施例1-11	A6	75	A3	25		53	54	75	100	96	25	12.0	12.1	16.0	19.8	20.0	33.8	72.3	3.6	0.7	<1.8
比較例1-15	A6	80	A3	20		53	45	76	103	98	20	12.5	9.8	16.8	22.9	20.8	32.9	73.1	3.6	0.7	4.1

[0058](Result) As shown in Table 2, about the spherical particle ratio of an aggregate particle, and Example 1-1 to 1-11 which has particle diameter within the limits of this invention 50%, both substrate flexural strength the amount of substrate water penetration and the maximum pole diameter of the filter layer showed the good result.

[0059]On the other hand, comparative example 1-1,1-2 whose spherical particle in an aggregate particle is more than 80 mass %, 1-4,1-6, 1-9,1-14, and 50% particle diameter all fell about comparative example 1-7,1-of more than 100 micrometers 8 as notably [substrate flexural strength] as 20 or less MPa.

[0060]As for comparative example 1-12,1-13 below 40 micrometers, particle diameter fell 50% as notably [the amount of substrate water penetration] as below 20-m^3/hr-m^2. About comparative example 1-3,1-5 whose spherical particle in an aggregate particle is below 20 mass %, and 1-15, there

was a tendency for the maximum pole diameter of a filter layer to become large. It is because the microstructure inside a substrate became uneven and it became uneven at the time of film production a slurry's adhering this to a substrate.

[0061]About Example 1-1 to 1-11 which has the particle size distribution of an aggregate particle within the limits of this invention, in the pole diameter of the substrate, d_{20} became 0.75 or more times of d_{50}, d_{80} had become 1.25 or less times of d_{50}, and pore volume distribution was sharp. On the other hand, even when a spherical particle ratio and 50% particle diameter were being filled, in the pore volume distribution of a substrate, d_{20} fell by comparative example 1-10,1-11 which does not have particle size distribution in the range of this invention, and pore volume distribution became broadcloth.

[0062](Example 2) Example 2 shows the example which produced the substrate like Example 1 by having made into the aggregate particle the mullite particles and SERUBEN particles which have various aspect ratios, 50% particle diameter, and particle size distribution, and also formed the filtration membrane, and was used as the filter.

[0063]

[Table 3]

	組成 骨材1 種類	%	骨材2 種類	%	骨材粒子 D50×0.7 μm	D20 μm	粒度分布 D50 μm	D90 μm	D50×1.3 μm	アスペクト比1.1以下粒子原料中比率 %	基材 D50×0.75 μm	平均細孔径 d20 μm	d50 μm	d90 μm	d50×1.25 μm	曲げ強度 MPa	透水量 m3/hr・m2	骨材粒子D50 μm	濾過層 中間層 μm	濾過層 濾過層 μm	濾過層 最大細孔径 μm
比較例2-1	M2	20	M1	80	52	63	74	86	96	80	12.3	13.6	16.4	19.1	20.5	18.3	71.5	-	-	-	-
実施例2-1	M2	25	M1	75	52	60	74	86	96	75	12.0	12.9	16.0	19.1	20.0	34.3	71.5	3.6	0.7	-	<1.8
実施例2-2	M2	50	M1	50	51	54	73	87	95	50	11.7	11.9	15.7	18.6	19.6	34.6	70.3	3.6	0.7	-	<1.8
実施例2-3	M2	75	M1	25	49	52	70	88	91	25	11.0	11.3	14.7	18.0	18.3	36.6	68.8	3.6	0.7	-	<1.8
比較例2-2	M2	80	M1	20	48	47	68	86	88	20	10.8	9.3	14.1	18.3	17.6	37.8	64.5	3.6	0.7	-	3.3
比較例2-3	M3	20	M1	80	53	65	76	87	99	80	12.1	13.6	16.1	18.2	20.1	18.1	73.6	-	-	-	-
実施例2-4	M3	25	M1	75	53	65	76	88	99	75	12.5	14.2	16.7	19.6	20.8	33.1	73.4	3.6	0.7	-	<1.8
実施例2-5	M3	50	M1	50	54	64	77	92	100	50	12.7	13.9	17.0	20.2	21.2	32.5	75.0	3.6	0.7	-	<1.8
実施例2-6	M3	75	M1	25	54	63	77	97	100	25	12.9	13.8	17.2	21.3	21.5	32.5	75.0	3.6	0.7	-	<1.8
比較例2-4	M3	80	M1	20	55	63	78	101	101	20	13.1	13.7	17.5	22.8	21.9	31.9	76.2	3.6	0.7	-	5.3
比較例2-5	S1	20	M1	80	52	64	74	87	96	80	12.4	14.1	16.5	19.2	20.6	18.3	71.5	-	-	-	-
実施例2-7	S1	25	M1	75	52	63	74	87	96	75	12.0	13.5	16.0	19.1	20.0	34.3	71.5	3.6	0.7	-	<1.8
実施例2-8	S1	50	M1	50	51	61	73	87	95	50	11.7	12.9	15.7	18.6	19.6	34.6	70.3	3.6	0.7	-	<1.8
実施例2-9	S1	75	M1	25	50	57	71	86	92	25	11.6	12.1	15.4	18.7	19.3	36.0	68.0	3.6	0.7	-	<1.8
比較例2-6	S1	80	M1	20	49	56	70	86	91	20	11.4	11.8	15.2	19.2	19.0	36.6	65.8	3.6	0.7	-	4.8

[0064](Result) As shown in Table 3, about the spherical particle ratio of an aggregate particle, and Example 2-1 to 2-9 which has particle diameter within the limits of this invention 50%, both substrate flexural strength the amount of substrate water penetration and the maximum pole diameter of the filter layer showed the good result.

[0065]On the other hand, about comparative example 2-1,2-3 whose spherical particle in an aggregate particle is more than 80 mass %, and 2-5, substrate flexural strength all fell notably with 20 or less MPa. About comparative example 2-2,2-4 whose spherical particle in an aggregate particle is below 20 mass %, and 2-6, there was a tendency for the maximum pole diameter of a filter layer to all become large. It is because it originated in the microstructure inside a substrate being uneven and adhesion of a slurry in a substrate became uneven at the time of film production.

[0066]

[Effect of the Invention]Since the substrate for honeycomb filters of this invention controlled the 50%

particle diameter of an aggregate particle and the mass ratio of a spherical particle which constitute a substrate within the limits of predetermined, its mechanical strength is high and its fluid transmission quantity is large. When the particle size distribution of an aggregate particle is controlled within the limits of predetermined, the pore volume distribution of a substrate will also become sharp.

[Translation done.]

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1]It is a schematic diagram showing a general structure of a honeycomb filter, and is a perspective view of the whole filter.
[Drawing 2]It is a schematic diagram showing the example of the film production device used for the filtration forming-membranes method.
[Description of Notations]
1 [— Bolt,] — A porous base material, 2, 3 — A flange, 4 — O-ring, 5 6 [— The slurry for membrane formation 10 / — Piping, 11, 14 / — A valve, 12 / — The inner wall of through hole of a porous base material, 13 / — A vacuum pump, 15, 16 / — A pressure gauge, 17 / — A breakthrough, A / — A feed hopper, B / — An outlet, 21 / — A filter, 22 / — A substrate, 23 / — Cell.] — A vacuum chamber, 7 — A slurry pump, 8 — A storage kettle, 9

[Translation done.]

(19)日本国特許庁（ＪＰ）　(12)公開特許公報（Ａ）　(11)特許出願公開番号
特開2001－340718
（P2001－340718A）
(43)公開日　平成13年12月11日(2001.12.11)

(51)Int.Cl.[7]		識別記号	ＦＩ		テーマコード（参考）
Ｂ０１Ｄ	39/20		Ｂ０１Ｄ	39/20	Ｄ　４Ｄ０１９
	39/00			39/00	Ｂ　４Ｇ０１９
Ｃ０４Ｂ	35/622		Ｃ０４Ｂ	38/00	３０３Ｚ　４Ｇ０３０
	38/00	３０３		35/00	Ｃ

審査請求　未請求　請求項の数７　ＯＬ　（全９頁）

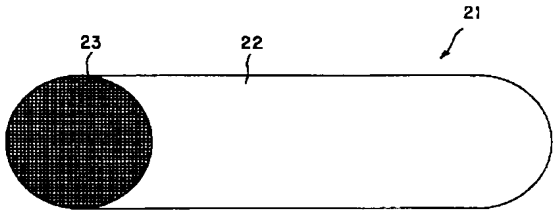
(21)出願番号　特願2000－165468(P2000－165468)
(22)出願日　平成12年６月２日(2000.6.2)

(71)出願人　000004064
日本碍子株式会社
愛知県名古屋市瑞穂区須田町２番56号
(72)発明者　馬場　龍夫
愛知県名古屋市瑞穂区須田町２番56号　日本碍子株式会社内
(74)代理人　100088616
弁理士　渡邊　一平

Ｆターム(参考)　4D019 AA01 AA03 BA05 BA06 BA07
BB06 BD01 BD10 CA01 CB06
4G019 FA01 FA12
4G030 AA36 AA37 BA20 CA09 CA10
GA11 GA14 GA21 HA04 HA15

(54)【発明の名称】　ハニカムフィルタ用基材及びその製造方法

(57)【要約】
【課題】　機械的強度が高く、流体透過量が大きいハニカムフィルタ用基材を提供する。
【解決手段】　多数のセル２３を有するハニカム構造の多孔体からなるハニカムフィルタ用の基材２２である。基材２２を構成する骨材粒子の５０％粒子径（D_{50}）を４０～１００μmの範囲内とし、かつ、骨材粒子の２０質量%超、８０質量%未満はアスペクト比１．１以下の球状粒子、残部はアスペクト比が球状粒子の１．２倍以上の非球状粒子から構成する。



1

【特許請求の範囲】

【請求項１】　多数のセルを有するハニカム構造の多孔体からなるハニカムフィルタ用の基材であって、
当該基材を構成する骨材粒子の５０％粒子径（D_{50}）が４０～１００μｍの範囲内にあり、かつ、当該骨材粒子の２０質量％超、８０質量％未満はアスペクト比１．１以下の球状粒子、残部はアスペクト比が前記球状粒子の１．２倍以上の非球状粒子からなることを特徴とするハニカムフィルタ用基材。

【請求項２】　骨材粒子の粒度分布が下記式（１）及び下記式（２）の関係を満たす請求項１に記載のハニカムフィルタ用基材。
$0.7 \times D_{50} \leqq D_{20}$　…（１）
$D_{80} \leqq 1.3 \times D_{50}$　…（２）
（但し、D_{20}：２０％粒子径、D_{50}：５０％粒子径、D_{80}：８０％粒子径）

【請求項３】　多数のセルを有するハニカム構造の多孔体からなるハニカムフィルタ用の基材であって、
５０％細孔径（d_{50}）が５～２５μｍの範囲内にあり、かつ、水銀圧入法により測定した細孔径分布が下記式（３）及び下記式（４）の関係を満たすハニカムフィルタ用基材。
$0.75 \times d_{50} \leqq d_{20}$　…（３）
$d_{80} \leqq 1.25 \times d_{50}$　…（４）
（但し、d_{20}：２０％細孔径、d_{50}：５０％細孔径、d_{80}：８０％細孔径）

【請求項４】　骨材粒子を含む坏土を、ハニカム構造と相補的な形状を有する押出用口金から押し出すことにより成形する工程を含むハニカムフィルタ用基材の製造方法であって、
５０％粒子径（D_{50}）が４０～１００μｍの範囲内にあり、かつ、アスペクト比が１．１以下である球状粒子の比率が２０質量％超、８０質量％未満、残部はアスペクト比が前記球状粒子の１．２倍以上である非球状粒子からなる骨材粒子から調製した坏土を使用することを特徴とするハニカムフィルタ用基材の製造方法。

【請求項５】　球状粒子を噴霧乾燥法により得る請求項４に記載のハニカムフィルタ用基材の製造方法。

【請求項６】　坏土を調製する骨材粒子の粒度分布が、下記式（１）及び下記式（２）の関係を満たす請求項４又は５に記載のハニカムフィルタ用基材の製造方法。
$0.7 \times D_{50} \leqq D_{20}$　…（１）
$D_{80} \leqq 1.3 \times D_{50}$　…（２）
（但し、D_{20}：２０％粒子径、D_{50}：５０％粒子径、D_{80}：８０％粒子径）

【請求項７】　請求項１～３のいずれか一項に記載のハニカムフィルタ用基材のセル内周面に、セル内周面に比して５０％細孔径が小さい濾過膜を少なくとも１層備えたハニカムフィルタ。

【発明の詳細な説明】

2

【０００１】
【発明の属する技術分野】　本発明は多数のセルを有するハニカム構造の多孔体からなるハニカムフィルタ用基材に関し、詳しくは機械的強度が高く、流体透過量が大きいハニカムフィルタ用基材に関する。

【０００２】
【従来の技術】　ハニカムフィルタは、例えば図１に示すような多数のセル２３を有するハニカム構造の多孔体を基材２２とするフィルタであり、多数のセル２３に供給されたガス、液体等の被処理流体が多孔体の細孔を透過する際に濾過が行われるため、単位体積あたりの濾過面積が大きい集塵フィルタ、固液分離フィルタとして利用されている。

【０００３】　例えば、フィルタ２１をハウジング内に収容し、基材２２の外周面側と端面側とを○－リング等で気密的に隔離する構造とすると、セル２３内に供給された被処理流体のうち基材の細孔内を透過した濾過流体のみを外周面側から流出させ、濾過されなかった被処理流体を端面側から回収するクロスフローフィルタとして利用することができる。

【０００４】　ハニカムフィルタにおいては、セル２３の内周面に、基材の細孔に比して更に細孔径が小さい濾過膜（０．０１～１．０μｍ程度）を少なくとも１層備える構造とし、基材内部の細孔径を極力大きく構成することが理想的である。このような構造では、細孔径が小さい濾過膜により濾過性能を確保する一方、細孔径が大きく（１～数１００μｍ程度）、内部の流動抵抗が低い基材により流体透過量を増加させることが期待できるからである。

【０００５】　一般に、ハニカムフィルタ用の基材は、骨材粒子を含む坏土を、ハニカム構造と相補的な形状を有する押出用口金から押し出すことにより成形する方法により製造される。従来、細孔径が大きい基材を得る方法としては、❶骨材粒子の粒径を大きくすることにより骨材粒子間の間隙部である細孔を大きくする方法（以下「第１の方法」という。）、或いは❷基材焼成時に焼失する有機物（ビッチ、コークス等）を坏土中に添加することにより、空隙部を形成させ通常と比較して細孔を大きくする方法（以下「第２の方法」という。）、等が採用されてきた。

【０００６】
【発明が解決しようとする課題】　しかしながら、第１の方法では、得られた基材が、フィルタとしての使用に耐え得る機械的強度を備えていないという問題があった。具体的には、基材のセル内周面に濾過膜を製膜する工程におけるハンドリングで破損するため製品歩留まりが低下したり、或いはフィルタとして使用する際に逆洗浄の圧力で破損する等の不具合があった。

【０００７】　また、第２の方法は、細孔径が大きくなり難く透水量が増加し難いことに加えて、基材焼成時に

有機物が急激に燃焼するため、熱衝撃により基材にクラックを生じるという問題があった。

【０００８】　即ち、従前においては、機械的強度が高く、流体透過量が大きいハニカムフィルタ用の基材は存在しておらず、そのような基材が切望されている。本発明は、このような従来技術の問題点に鑑みてなされたものであって、その目的とするところは、機械的強度が高く、流体透過量が大きいハニカムフィルタ用基材を提供することにある。

【０００９】

【課題を解決するための手段】　本発明者らが鋭意検討した結果、基材を構成する骨材粒子の５０％粒子径及び球状粒子の質量比を所定の範囲内に制御することにより、従来技術の問題点を解決できることに想到して本発明を完成した。

【００１０】　即ち、本発明によれば、多数のセルを有するハニカム構造の多孔体からなるハニカムフィルタ用の基材であって、当該基材を構成する骨材粒子の５０％粒子径（D_{50}）が４０～１００μｍの範囲内にあり、かつ、当該骨材粒子の２０質量%超、８０質量%未満はアスペクト比１．１以下の球状粒子、残部はアスペクト比が前記球状粒子の１．２倍以上の非球状粒子からなることを特徴とするハニカムフィルタ用基材が提供される。

【００１１】　上記ハニカムフィルタ用基材は、骨材粒子の粒度分布が下記式（１）及び下記式（２）の関係を満たすことが好ましい。

$0.7 \times D_{50} \leqq D_{20}$　…（１）

$D_{80} \leqq 1.3 \times D_{50}$　…（２）

（但し、D_{20}：２０％粒子径、D_{50}：５０％粒子径、D_{80}：８０％粒子径）

【００１２】　また、本発明によれば、多数のセルを有するハニカム構造の多孔体からなるハニカムフィルタ用の基材であって、５０％細孔径（d_{50}）が５～２５μｍの範囲内にあり、かつ、水銀圧入法により測定した細孔径分布が下記式（３）及び下記式（４）の関係を満たすハニカムフィルタ用基材が提供される。

$0.75 \times d_{50} \leqq d_{20}$　…（３）

$d_{80} \leqq 1.25 \times d_{50}$　…（４）

（但し、d_{20}：２０％細孔径、d_{50}：５０％細孔径、d_{80}：８０％細孔径）

【００１３】　更に、本発明によれば、骨材粒子を含む坏土を、ハニカム構造と相補的な形状を有する押出用口金から押し出すことにより成形する工程を含むハニカムフィルタ用基材の製造方法であって、５０％粒子径（D_{50}）が４０～１００μｍの範囲内にあり、かつ、アスペクト比が１．１以下である球状粒子の比率が２０質量%超、８０質量%未満、残部はアスペクト比が前記球状粒子の１．２倍以上である非球状粒子からなる骨材粒子から調製した坏土を使用することを特徴とするハニカムフィルタ用基材の製造方法が提供される。

【００１４】　本発明の製造方法においては、球状粒子を噴霧乾燥法により得ることが好ましく、坏土を調製する骨材粒子の粒度分布が、下記式（１）及び下記式（２）の関係を満たすことが好ましい。

$0.7 \times D_{50} \leqq D_{20}$　…（１）

$D_{80} \leqq 1.3 \times D_{50}$　…（２）

（但し、D_{20}：２０％粒子径、D_{50}：５０％粒子径、D_{80}：８０％粒子径）

【００１５】　更にまた、本発明によれば、上記のハニカムフィルタ用基材のセル内周面に、セル内周面に比して５０％細孔径が小さい濾過膜を少なくとも１層備えたハニカムフィルタが提供される。

【００１６】

【発明の実施の形態】　本発明のハニカムフィルタ用基材は、基材を構成する骨材粒子の５０％粒子径及び球状粒子の質量比を所定の範囲内に制御したものである。本発明のハニカムフィルタ用基材は、機械的強度が高く、流体透過量が大きい。以下、本発明について詳細に説明する。

【００１７】（１）基材

本発明のハニカムフィルタ用基材（以下、単に「基材」という。）は、骨材粒子のうち少なくとも１種をアスペクト比が１．１以下である球状粒子としたものである。このような球状粒子を骨材粒子とすることにより、基材焼成後においても骨材粒子間に確実に間隙部（即ち細孔）が形成され、流体の透過量を大きくすることができる。具体的には、前記球状粒子を骨材粒子中に２０質量%以上含むことにより流体透過量を増加させ、基材内部の微構造を均一化する効果を得ることができる。

【００１８】　但し、基材を構成する骨材粒子の全てを上記球状粒子とすると骨材粒子間の結合が弱くなり、基材の機械的強度が低下する。従って、前記球状粒子は骨材粒子の２０質量%以上とすることに加え、その上限を８０質量%とする必要がある。

【００１９】　骨材粒子のうち球状粒子以外の残部についてはアスペクト比が上記球状粒子の１．２倍以上である非球状粒子により構成する。即ち、球状粒子としてアスペクト比１．０の粒子を用いた場合には１．２以上、１．１のものを用いた場合であれば１．３２以上のアスペクト比を有する粒子が非球状粒子となる。非球状粒子は流体透過量を増加させるという観点からは好ましくないが、骨材粒子間の結合を強化する効果を有し、基材の機械的強度を向上させる作用がある。

【００２０】　なお、本発明にいう「アスペクト比」とは、粉末状態においては走査型電子顕微鏡で撮影した写真から任意に選択した２０個の骨材粒子のアスペクト比（長辺と短辺の比）の平均値、焼結体においては、焼結体の切断面に樹脂（例えばエポキシ樹脂）を塗布して穴埋めし、更に鏡面仕上げを行った面について同様に算出したアスペクト比の平均値である。

【００２１】　球状粒子、非球状粒子は、既述のアスペクト比を満たしていることに加え、５０％粒子径（D_{50}）が所定の範囲、具体的には４０～１００μｍの範囲内に制御されていることが必要である。５０％粒子径（D_{50}）が４０μｍ未満の場合には流体透過量が減少する点において、１００μｍ超となる場合には基材の機械的強度が低下する点においていずれも好ましくない。また、５０％粒子径（D_{50}）を４０～１００μｍの範囲内とすることにより、基材の５０％細孔径（d_{50}）も５～２５μｍ（水銀圧入法）の範囲内に制御される。

【００２２】　なお、本発明に言う「ｘ％粒子径」とは、粉末状態においては篩分け法により測定した粒子径である。具体的には、公称目開き径の異なる複数の篩を、上段ほど目開き径が大きくなるように多段に積重したものを用意し、最上段の篩に粒子径の測定対象である粉体試料を注入し、振とう機で１５分間振とうした後、各段の篩上にある粉末質量とその篩の目開き径との関係から粒度分布曲線を作成し、積算質量がｘ％となる粒子径をｘ％粒子径と規定した。

【００２３】　球状粒子、非球状粒子は、既述の球状粒子比率、５０％粒子径（D_{50}）を満たしていることに加え、粒度分布が所定の範囲に制御されていること、具体的には下記式（１）及び下記式（２）の関係を満たすことが好ましい。

$0.7 \times D_{50} \leqq D_{20}$　…（１）

$D_{80} \leqq 1.3 \times D_{50}$　…（２）

（但し、D_{20}：２０％粒子径、D_{50}：５０％粒子径、D_{80}：８０％粒子径）

【００２４】　骨材粒子の粒度分布が上記式（１）及び上記式（２）の関係を満たす基材は細孔径分布がシャープとなるからである。具体的には、水銀圧入法により測定した細孔径分布が下記式（３）及び下記式（４）の関係を満たす範囲内に制御された基材となる。

$0.75 \times d_{50} \leqq d_{20}$　…（３）

$d_{80} \leqq 1.25 \times d_{50}$　…（４）

（但し、d_{20}：２０％細孔径、d_{50}：５０％細孔径、d_{80}：８０％細孔径）

【００２５】　一方、上記式（１）を満たさない場合には微粒成分が多くなることに起因して基材の骨材粒子間の空隙部が閉塞され、流体透過量が減少するおそれがある。また、上記式（２）を満たさない場合には、骨材粒子間の空隙部は大きくなるが、製膜工程において製膜用スラリーが当該空隙部に入り込み閉塞するため、やはりフィルタの流体透過量は低下するおそれがある。

【００２６】　なお、本発明にいう「ｘ％細孔径」とは、下記式（５）を原理式とする水銀圧入法により測定した細孔径である。具体的には、乾燥した基材に対して徐々に圧力を上昇させながら水銀を圧入すると、径の大きい細孔から順に水銀が圧入されて水銀の累積容量が増加していき、最終的に全ての細孔が水銀で満たされる

と、累積容量は衡量に達する。本発明においては、累積容量がｘ％となった際の圧力Ｐから算出された細孔径ｄをｘ％細孔径と規定した。

$d = -\gamma \times \cos\theta / P$　…（５）

（但し、ｄ：細孔径、γ：表面張力、θ：接触角、Ｐ：圧力）

【００２７】（２）製造方法

ハニカムフィルタ用の基材は、骨材粒子を含む坏土を、ハニカム構造と相補的な形状を有する押出用口金から押し出すことにより成形し、当該成形体を乾燥し、焼成することにより得られる。本発明の基材を製造するためには、坏土を調製する際にアスペクト比、５０％粒子径、粒度分布を既述の範囲内に制御した骨材粒子を使用すればよい。

【００２８】　即ち、５０％粒子径（D_{50}）が４０～１００μｍの範囲内にあり、かつ、アスペクト比が１．１以下である球状粒子の比率が２０質量％超、８０質量％未満、残部はアスペクト比が前記球状粒子の１．２倍以上である非球状粒子からなる骨材粒子から坏土を調製する。基材の細孔径分布をシャープにしたい場合には、当該骨材粒子の粒度分布が下記式（１）及び下記式（２）の関係を満たすようにする。

$0.7 \times D_{50} \leqq D_{20}$　…（１）

$D_{80} \leqq 1.3 \times D_{50}$　…（２）

（但し、D_{20}：２０％粒子径、D_{50}：５０％粒子径、D_{80}：８０％粒子径）

【００２９】　調製の方法としては、例えば市販のセラミック原料をそのまま、或いはこれを粉砕・分級したものを骨材粒子とし、２種以上の骨材粒子を既述の条件を満たすように適宜混合する方法などが挙げられる。

【００３０】　本発明の製造方法においては、球状粒子を噴霧乾燥法により得ることが好ましい。球状粒子は粉砕機や混合機（ボールミル等）で粉砕・混合する方法により調製したものを使用しても良いが、液状とした原料をスプレードライヤーにより造粒・乾燥し、焼成する噴霧乾燥法によれば、比較的容易にアスペクト比１．１以下の球状粒子が得られるからである。また、噴霧乾燥法により得られた粒子は粉砕により得られた粒子と比較して表面が平滑であるため、押出成形用口金を痛めずその耐用期間が１０倍程度に長くなる点においても好ましい。

【００３１】　本発明の製造方法は、坏土を調製する際にアスペクト比、５０％粒子径、所望により粒度分布を既述の範囲内に制御した骨材粒子を使用することを除き、従来公知の製造方法と同様の方法により製造することが可能である。

【００３２】　坏土は、骨材粒子の他、分散媒、有機バインダ、必要により無機結合材、界面活性剤、可塑剤等を添加し、混練し成形原料とする。

【００３３】　骨材粒子としては、アルミナ、ムライ

ト、セルベン、コージェライト、炭化珪素或いはこれらの混合物等を、分散媒としては、水等を、有機バインダとしてはメチルセルロース等を用いることができる。

【００３４】　無機結合材は、骨材粒子の結合を強化するための添加材であり、粒径１μｍ未満のアルミナ、シリカ、ジルコニア、チタニア、ガラスフリット、長石、コージェライトのうちの１種又は２種以上の混合物を使用することができる。なお、無機結合材はセラミック粒子ではあるが本発明にいう骨材粒子には包含されない。

【００３５】　無機結合材は、骨材粒子の質量を１００質量%とした場合において、これに対し、１５質量%以上、３５質量%以下に相当する量を添加することが好ましい。１５質量%未満であると基材の強度が低下する一方、３５質量%超となると充分な強度は得られるものの骨材粒子の間隙に無機結合材が止まるため、基材内部の細孔を閉塞し流体透過量を低下させるおそれがあるからである。

【００３６】　坏土を所望の形状に押出成形し、乾燥・焼成することによりハニカム構造の基材を製造することができる。例えば、単軸、２軸、或いは多軸のスクリュー押出機やプランジャー押出機等の従来公知の押出機に投入した坏土を、基材のハニカム構造と相補的な形状を有する押出用口金から押し出すことにより成形体を得ることができる。

【００３７】　口金の形状により、基材の端面形状（円形、正方形、長方形、六角形等）、端面外径（円形の場合３０～２００ｍｍφ）、セルの形状（円形、四角形、六角形等）、セルの内接孔直径（通常は２～５ｍｍφ程度）等を所望の形状とすることが可能である。基材のサイズは特に限定されないが、長手方向の全長が１５０～２０００ｍｍ程度のものが汎用される。

【００３８】（３）フィルタ

上述の基材のセル内周面に、骨材粒子を含む製膜用スラリーを付着せしめた後、当該製膜体を乾燥・焼成する方法により濾過膜を形成することができ、ハニカムフィルタを得ることができる。

＊【００３９】　例えば、骨材粒子を水等の分散媒中に分散し、必要に応じ有機バインダ、ｐＨ調整剤、界面活性剤等を添加することにより製膜用のスラリーとし、従来公知の方法、例えばディップ製膜法、本出願人が既に開示した特公昭６３－６６５６６号公報に記載の濾過製膜法等を用いてセル内周面に成膜して乾燥し、更に当該製膜体を１３００℃程度の高温で焼成する等の方法によりフィルタを得ることができる。骨材粒子、分散媒、有機バインダについては基材と同様のものを使用することができる。但し、濾過膜の細孔径を小さくするため骨材粒子の５０%粒子径は基材よりも小さくすることが一般的である。

【００４０】　また、製膜用スラリーには基材と同様の目的で無機結合材を含有させても良い。濾過膜の場合には、粒径１μｍ未満の粘土、カオリン、チタニアゾル、シリカゾル、ガラスフリット等を用いることができ、骨材粒子及び無機結合材の全質量中に、５質量%以上、２５質量%以下の比率で含まれていることが好ましい。

【００４１】　なお、濾過膜は少なくとも１層形成することが必要があるが、２層以上形成して複層としてもよい。

【００４２】

【実施例】　以下、本発明のフィルタを実施例により更に詳細に説明するが、本発明は下記の実施例により限定されるものではない。

【００４３】　基材の骨材粒子となるセラミック原料としては、表１に記載の組成及びアスペクト比を有するアルミナ（Ａ１～Ａ６），ムライト（Ｍ１～Ｍ３），セルベン（Ｓ１）を使用した。これらの原料を表２～３に記載の比率で混合して骨材粒子を調製した。

【００４４】　なお、Ａ３，Ｍ１については、原料を液状とした後スプレードライヤーにより造粒・乾燥し、焼成することによりアスペクト比を１．１以下とした。

【００４５】

【表１】

＊

記号	種類	粒度分布					アスペクト比	
		D50×0.7 μm	D20 μm	D50 μm	D80 μm	D50×1.3 μm	平均	1.1以下粒子 %
A1	アルミナ	60	62	85	106	111	1.8	0
A2		49	55	70	86	91	1.9	0
A3		53	65	75	85	98	1.1	100
A4		74	92	105	135	137	1.8	0
A5		18	18	26	33	34	1.8	0
A6		53	41	75	105	98	1.7	0
M1	ムライト	53	65	76	85	99	1	100
M2		46	45	65	87	85	1.5	0
M3		55	62	78	104	101	1.8	0
S1	セルベン	49	54	70	87	91	1.8	0

【００４６】（基材）上記骨材粒子に、無機結合材（長石、ガラスフリット等）、水の他、有機バインダとしてメチルセルロースを加えて混練した坏土を押出成形し、外径φ３０ｍｍ、直径φ２．４ｍｍのセルを３７本有す

るハニカム構造の押出成形体を得た。無機結合材は骨材粒子の質量を１００質量％とした場合において、これに対し２５質量％に相当する量を添加した。当該押出成形体を電気炉で１５００℃で焼成することにより基材を得た。

【００４７】　上記基材は、基材の５０％細孔径及び細孔径分布、機械的強度について評価した。

【００４８】　基材の５０％細孔径及び細孔径分布については、水銀圧入法に従って以下の方法により測定した。まず、基材を端面から２５ｍｍの長さだけ切り出し、更にセル４～５個残るように切削して測定用サンプルとし、当該サンプルを水銀中に浸漬した状態で水銀を圧入し、その累積容量を測定することにより２０％細孔径、５０％細孔径、８０％細孔径を算出した。

【００４９】　基材の機械的強度については、各成形用坏土を直径φ２０ｍｍ×長さ１００ｍｍの円筒状に押出した成形体を、基材と同様の条件で乾燥し、焼成してなる焼結体を支点間距離８０ｍｍとして３点曲げ強度の試験を行うことにより評価した。

【００５０】　上記基材は濾過膜を形成してフィルタとしての性能についても評価した。濾過膜は複層構造とし、中間層と濾過層を設けた。

【００５１】　中間層及び濾過層の製膜は、特開昭６１－２３８３１５号公報に記載の濾過成膜法により行った。具体的には、図２に示す真空チャンバ６、貯蔵槽８、ポンプ７、フランジ２，３、配管１０等からなる装置に対し、細孔内を水などの液体で置換した基材１のセル内周面側と基材１外周面側とをフランジ２，３，ボルト５で気密的に隔離した状態で固定し、次いで貯蔵槽８内のスラリー９をポンプ７により基材１のセル内に連続的に送液してセル内周面１２に接触させながら、真空チャンバ６内を真空ポンプ１３により真空排気し、基材１外周面側を減圧状態とする。このような操作により、基材１外周面側とセル内周面１２側との間に濾過差圧が付与されるため、基材１のセル内周面１２にはスラリーが製膜され、スラリー中の水分は濾液として基材１外周面側から排出される。

【００５２】（中間層）基材と同材質で５０％粒子径（D_{50}）が３．２μｍの骨材粒子、無機結合材（長石、ガラスフリット等）、水を２７：３：７０の質量比で混合してスラリーを調製した。各セルの内周面にスラリーを製膜した後、乾燥し、焼成することにより基材に固着させ中間膜を形成した。

【００５３】（濾過層）基材と同材質で５０％粒子径（D_{50}）が０．４μｍの骨材粒子、無機結合材（ガラスフリット）、水を９：１：９０の質量比で混合してスラリーを調製した。各セルの中間層の表面にスラリーを製膜した後、乾燥し、焼成することにより中間層に固着させ濾過層を形成した。

【００５４】　上記のように製造されたフィルタについては、流体透過量を透水量により、濾過性能を濾過層の最大細孔径により評価した。透水量は、水中、６．７ｋＰａ以下の減圧下で２時間、フィルタ内の気泡を脱気した後、差圧４．８～９．８ｋＰａ、温度２５℃の条件で純水をフィルタのセルに注入し、セル内からフィルタ外周面側へ、透過させることにより濾過し、濾過面積当たりの時間当たりの透水量を測定することにより評価した。

【００５５】　濾過層の最大細孔径については以下の方法で測定した。ＡＳＴＭ　Ｆ３１６に記載のエアフロー法に準拠し、水温２０℃の水で湿潤したフィルタに対し、圧力を徐々に上昇させながら加圧エアをセル内周面から送り込み、基材外周面から最初に気泡が確認された圧力Ｐから算出された細孔径Ｄを濾過層の最大細孔径とした。

【００５６】（実施例１）実施例１として、種々の球状粒子比率、５０％粒子径、粒度分布を有するアルミナ粒子を骨材粒子として基材を作製し、更に濾過膜を形成してフィルタとした例を示す。

【００５７】

【表２】

11　　12

	骨材1 種類	骨材1	組成 骨材2 種類		骨材粒子 D50×0.7	骨材粒子 D20	骨材粒子 粒度分布 D50	骨材粒子 D90	骨材粒子 D50×1.3	アスペクト比1.1以下粒子原料中比率	基材 平均細孔径 d50×0.75	基材 平均細孔径 d20	基材 平均細孔径 d50	基材 平均細孔径 d80	基材 d50×1.25	曲げ強度	透水量	濾過層 骨材粒子D50 中間層	濾過層 骨材粒子D50 濾過層	濾過層 最大細孔径
		%		%	μm	μm	μm	μm	μm	%	μm	μm	μm	μm	μm	MPa	m3/hr·m2	μm	μm	μm
比較例1-1	A1	0	A3	100	53	65	75	85	96	100	12.2	13.9	16.3	18.4	20.4	18.5	72.7	-	-	-
比較例1-2	A1	20	A3	80	53	64	75	86	96	80	12.4	13.9	16.5	18.7	20.7	18.7	72.7	-	-	-
実施例1-1	A1	25	A3	75	53	63	76	88	99	75	12.5	13.9	16.7	19.6	20.8	33.1	73.8	3.6	0.7	<1.8
実施例1-2	A1	50	A3	50	54	62	77	92	100	50	12.7	13.7	17.0	20.2	21.2	32.5	75.0	3.6	0.7	<1.8
実施例1-3	A1	75	A3	25	55	62	78	97	101	25	13.0	13.0	17.3	21.2	21.7	31.9	76.2	3.6	0.7	<1.8
比較例1-3	A1	80	A3	20	55	61	78	101	101	20	13.2	13.2	17.6	22.9	22.0	31.9	76.2	3.6	0.7	4.2
比較例1-4	A2	20	A3	80	52	63	74	85	96	80	12.1	13.5	16.1	18.3	20.1	19.3	71.5	-	-	-
実施例1-4	A2	25	A3	75	52	62	74	86	96	75	12.0	13.3	16.0	18.9	20.0	34.3	71.5	3.6	0.7	<1.8
実施例1-5	A2	50	A3	50	51	60	73	86	95	50	11.7	12.7	15.7	18.4	19.6	34.8	70.3	3.6	0.7	<1.8
実施例1-6	A2	75	A3	25	50	57	71	87	92	25	11.2	11.7	15.0	18.0	18.7	36.0	68.0	3.6	0.7	<1.8
比較例1-5	A2	80	A3	20	49	56	70	87	91	20	11.0	11.4	14.7	18.4	18.4	38.6	66.8	3.6	0.7	2.8
比較例1-6	A4	20	A3	80	53	67	76	96	99	80	12.8	14.9	17.1	21.2	21.4	18.1	73.8	-	-	-
実施例1-7	A4	25	A3	75	55	70	78	99	101	75	13.0	15.5	17.3	20.6	21.7	31.9	76.2	3.6	0.7	<1.8
実施例1-8	A4	50	A3	50	62	72	88	113	114	50	15.5	18.7	20.7	24.8	25.8	26.1	87.8	3.6	0.7	<1.8
比較例1-7	A4	75	A3	25	71	77	102	125	133	25	18.0	18.8	25.3	30.6	31.7	17.9	104.2	-	-	-
比較例1-8	A4	80	A3	20	74	85	105	128	137	20	18.7	20.9	26.3	32.2	32.9	18.2	107.7	-	-	-
比較例1-9	A5	20	A3	80	51	30	73	83	95	80	11.9	9.4	15.9	18.7	19.9	13.8	70.3	-	-	-
比較例1-10	A5	25	A3	75	50	26	72	80	94	75	11.5	8.1	15.3	18.6	19.2	35.4	69.2	3.6	0.7	3.8
比較例1-11	A5	50	A3	50	29	20	41	88	53	50	6.2	5.8	8.3	9.1	10.4	53.5	33.0	-	-	-
比較例1-12	A5	75	A3	25	19	19	27	63	35	25	4.1	3.9	5.5	6.5	6.9	61.7	18.7	-	-	-
比較例1-13	A5	80	A3	20	18	18	25	35	33	20	4.0	3.6	5.3	6.7	6.6	62.8	14.3	-	-	-
比較例1-14	A6	20	A3	80	53	63	75	86	98	80	12.1	13.3	16.1	18.3	20.1	18.7	71.1	-	-	-
実施例1-9	A6	25	A3	75	53	61	75	88	98	75	12.3	13.2	16.4	19.5	20.5	33.4	70.2	3.6	0.7	<1.8
実施例1-10	A6	50	A3	50	53	55	75	92	98	50	12.2	12.3	16.2	19.6	20.3	33.1	69.8	3.6	0.7	<1.8
実施例1-11	A6	75	A3	25	53	54	75	100	98	25	12.0	12.1	16.0	19.8	20.0	33.6	72.3	3.6	0.7	<1.8
比較例1-15	A6	80	A3	20	53	46	75	100	98	20	12.5	9.8	16.8	22.9	20.8	32.9	73.1	3.8	0.7	4.1

【００５８】（結果）表２に示したように、骨材粒子の球状粒子比率、５０％粒子径が本発明の範囲内にある実施例１－１～１－１１については、基材曲げ強度、基材透水量、濾過層の最大細孔径のいずれも良好な結果を示した。

【００５９】　一方、骨材粒子中の球状粒子が８０質量％以上である比較例１－１，１－２，１－４，１－６，１－９，１－１４、５０％粒子径が１００μm超の比較例１－７，１－８についてはいずれも基材曲げ強度が２０ＭＰａ以下と顕著に低下した。

【００６０】　また、５０％粒子径が４０μm未満の比較例１－１２，１－１３は基材透水量が２０m^3／ｈｒ・m^2以下と顕著に低下した。更に、骨材粒子中の球状粒子が２０質量％以下である比較例１－３，１－５，１－１５については濾過層の最大細孔径が大きくなる傾向があった。これは基材内部の微構造が不均一となり、製膜時において、基材へのスラリーの付着が不均一となったことによるものである。

【００６１】　更に、骨材粒子の粒度分布が本発明の範囲内にある実施例１－１～１－１１については、基材の

13

細孔径がd_{20}がd_{50}の０．７５倍以上、d_{80}がd_{50}の１．２５倍以下となっており、細孔径分布がシャープであった。一方、球状粒子比率と５０％粒子径を満たしている場合でも、粒度分布が本発明の範囲にない比較例１－１０，１－１１は基材の細孔径分布においてd_{20}が低下し、細孔径分布はブロードとなった。

【００６２】（実施例２）実施例２は、実施例１と同様にして、種々のアスペクト比、５０％粒子径、粒度分布を有するムライト粒子、セルベン粒子を骨材粒子として基材を作製し、更に濾過膜を形成してフィルタとした例を示す。

【００６３】

【表３】

14

	組成 骨材1 種類		組成 骨材2 種類		骨材粒子 D50×0.7	骨材粒子 粒度分布 D20	骨材粒子 粒度分布 D50	骨材粒子 粒度分布 D80	骨材粒子 粒度分布 D50×1.3	アスペクト比 1.1以下粒子 原料中比率	基材 平均細孔径 d50×0.75	基材 平均細孔径 d20	基材 平均細孔径 d50	基材 平均細孔径 d80	基材 平均細孔径 d50×1.25	基材 曲げ強度	基材 透水量	濾過膜 骨材粒子D50 中間層	濾過膜 骨材粒子D50 濾過膜	濾過膜 最大細孔径
		%		%	μm	μm	μm	μm	μm	%	μm	μm	μm	μm	μm	MPa	m3/hr-m2	μm	μm	μm
比較例2-1	M2	20	M1	80	52	63	74	86	96	80	12.3	13.6	16.4	19.1	20.5	19.3	71.5	-	-	-
実施例2-1	M2	25	M1	75	52	60	74	86	96	75	12.0	12.9	16.0	19.1	20.0	34.3	71.5	3.6	0.7	<1.6
実施例2-2	M2	50	M1	50	51	54	73	87	95	50	11.7	11.9	15.7	18.6	19.6	34.6	70.3	3.6	0.7	<1.6
実施例2-3	M2	75	M1	25	49	52	70	88	91	25	11.0	11.3	14.7	18.0	18.3	36.6	68.8	3.6	0.7	<1.6
比較例2-2	M2	80	M1	20	48	47	68	88	88	20	10.8	9.3	14.1	18.3	17.6	37.8	64.5	3.6	0.7	3.3
比較例2-3	M3	20	M1	80	53	65	78	87	99	80	12.1	13.6	16.1	18.2	20.1	18.1	73.8	-	-	-
実施例2-4	M3	25	M1	75	53	65	76	88	99	75	12.5	14.2	16.7	19.6	20.8	33.1	73.8	3.6	0.7	<1.6
実施例2-5	M3	50	M1	50	54	64	77	92	100	50	12.7	13.9	17.0	20.2	21.2	32.5	75.0	3.6	0.7	<1.6
実施例2-6	M3	75	M1	25	54	63	77	97	100	25	12.8	13.6	17.2	21.3	21.5	32.5	75.0	3.6	0.7	<1.6
比較例2-4	M3	80	M1	20	55	63	78	101	101	20	13.1	13.7	17.5	22.8	21.9	31.9	78.2	3.6	0.7	5.3
比較例2-5	S1	20	M1	80	52	64	74	87	96	80	12.4	14.1	16.5	19.2	20.6	19.3	71.5	-	-	-
実施例2-7	S1	25	M1	75	52	63	74	87	96	75	12.0	13.5	16.0	19.1	20.0	34.3	71.5	3.6	0.7	<1.6
実施例2-8	S1	50	M1	50	51	61	73	87	95	50	11.7	12.9	15.7	18.6	19.6	34.6	70.3	3.6	0.7	<1.6
実施例2-9	S1	75	M1	25	50	57	71	88	92	25	11.8	12.1	15.4	18.7	19.3	36.0	68.0	3.6	0.7	<1.6
比較例2-6	S1	80	M1	20	49	56	70	88	91	20	11.4	11.8	15.2	19.2	19.0	36.6	66.8	3.6	0.7	4.8

【００６４】（結果）表３に示したように、骨材粒子の球状粒子比率、５０％粒子径が本発明の範囲内にある実施例２－１～２－９については、基材曲げ強度、基材透水量、濾過層の最大細孔径のいずれも良好な結果を示した。

【００６５】　一方、骨材粒子中の球状粒子が８０質量％以上である比較例２－１，２－３，２－５についてはいずれも基材曲げ強度が２０ＭＰａ以下と顕著に低下した。骨材粒子中の球状粒子が２０質量％以下である比較例２－２，２－４，２－６についてはいずれも濾過層の

15

最大細孔径が大きくなる傾向があった。基材内部の微構造が不均一であることに起因して、製膜時において、基材へのスラリーの付着が不均一となったためである。

【００６６】

【発明の効果】　本発明のハニカムフィルタ用基材は、基材を構成する骨材粒子の５０％粒子径及び球状粒子の質量比を所定の範囲内に制御したので、機械的強度が高く、流体透過量が大きい。また、骨材粒子の粒度分布を所定の範囲内に制御した場合には基材の細孔径分布もシャープなものとなる。

【図面の簡単な説明】

【図１】　ハニカムフィルタの一般的な構造を示す概略＊

16

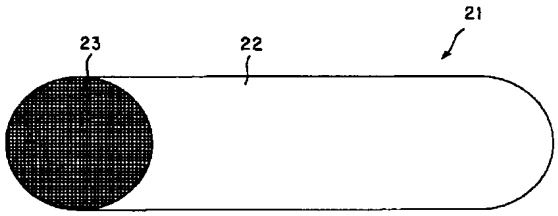
＊図であって、フィルタ全体の斜視図である。

【図２】　濾過成膜法に使用する製膜装置の例を示す概略図である。

【符号の説明】

１…多孔質基材、２，３…フランジ、４…Ｏ－リング、５…ボルト、６…真空チャンバ、７…スラリーポンプ、８…貯蔵槽、９…成膜用スラリー、１０…配管、１１，１４…バルブ、１２…多孔質基材の貫通孔内壁、１３…真空ポンプ、１５，１６…圧力計、１７…貫通孔、Ａ…供給口、Ｂ…排出口、２１…フィルタ、２２…基材、２３…セル。

【図１】



【図２】